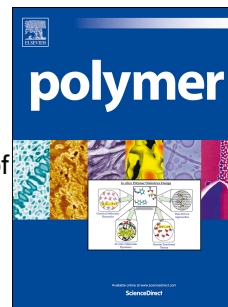


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Altering the Effectiveness of Radical Traps in Atom Transfer Radical Coupling
Reactions of Polymer Chains

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ABSTRACT: Atom transfer radical coupling (ATRC) reactions assisted by radical traps were studied under various conditions, with the extent of coupling of polymer chains monitored as a function of time. When using a radical trap that tended to aggregate into dimers, such as 2-methyl-2-nitrosopropane (MNP), the rate of coupling was enhanced when conditions were adjusted to favor its deaggregation to the monomeric form: less polar solvent mixtures and/or the addition of β -cyclodextrin (β -CD). With radical traps that did not aggregate, such as nitrosobenzene (NBz) and *N-tert*-Butyl- α -phenylnitrone (tBuPN), coupling was generally accelerated under conditions consistent with higher K_{ATRP} values. MALDI-TOF analysis was used to confirm the incorporation of the radical trap into the polymer dimers, supporting the mechanistic pathway proposed for radical trap-assisted ATRC. The effect of aggregation of the radical trap was further probed by using fractional equivalents of NBz in the coupling reaction, with a full equivalent leading to end-capped polymer chains that were unable to dimerize. By reducing the equivalents of NBz, the extent of coupling could be controlled by the temperature, with higher temperatures (80 °C) allowing ATRC to compete with RTA-ATRC and higher extents of coupling were observed.

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